

Appln. No. 10/508,805  
Response B dated August 29, 2007  
Reply to Office Action of June 7, 2007

### REMARKS/ARGUMENTS

#### A. Regarding the Amendments

Claims 7 to 9 and 11 to 51 remain pending in the application. Claim 10 is canceled herein. Claims 1 to 6 were canceled in Applicants' Response to the Final Rejection, filed February 8, 2007.

Claims 8 and 9 are amended to delete subject matter related to the process of making the composition.

Claims 11, 24, 33, and 39 are amended to require the metathesis catalyst to achieve "a turnover number greater than 1,500." Support for this amendment is found at page 30, lines 1-6, of the specification.

Claims 11, 17, 18, 24, 25, 26, 33, 34, 39, and 40 are amended to require the unsaturated fatty acid/ester feedstock to comprise "less than 25 meq metathesis catalyst poisons per kg composition." Support for this amendment is found at page 20, lines 24-27, of the specification. This amendment relates to a preferred lower level of metathesis poisons for achieving a catalyst turnover number greater than 1,500.

Claims 11, 21, 24, 27, 33, 35, 39, and 41 are further amended to replace the term "lower olefin" with its definition "C<sub>2-3</sub> olefin". Support for this amendment is found at page 22, lines 14-15, of the specification. This amendment is made for clarification as well as for the fact that C<sub>2-3</sub> olefins are preferred in achieving a catalyst turnover number greater than 1,500.

Claim 12 is amended to delete the number "25" in relation to "meq hydroperoxides per kg feedstock composition." Substituted therefor is the number "15." Support for this amendment is found at page 20, lines 24-27, of the specification.

Claim 21 is amended to limit the C<sub>2-3</sub> olefin to "ethylene." Support for this amendment is found at page 22, lines 14-15, of the specification.

Applicants estimate that no fee is required for the aforementioned amendments. If this estimate is incorrect, the Examiner is authorized to charge Debit Account 04-1512 the required fee.

Appl. No. 10/508,805  
Response B dated August 29, 2007  
Reply to Office Action of June 7, 2007

**B. Regarding the Objection to Claims 7-10**

Claims 7 to 10 are objected to for being drawn to compounds in the context of a product-by-process format. The Office Action asserts that the compounds themselves are definite; whereas product-by-process format is reserved for compositions that cannot be claimed in a definite matter.

Applicants note that Claim 7 has not been drafted in product-by-process format; however, with respect to Claims 8-10, the point is conceded. Claims 8 and 9 are amended herein to delete process limitations, and Claim 10 is canceled. The novel and unobvious properties of the claimed compositions are discussed hereinbelow. In view of the above, it is respectfully requested that the objection be withdrawn.

**C. Regarding the Rejection of Claims 7-10, and 45-48 Under 35 USC 102(b)**

Claims 7-10 and 45-48 stand rejected under 35 USC 102(b) as allegedly being anticipated with reference to Examples 1, 3, and 4 of DE 4,107,056 and the examples at page 17 of WO 96/04289. This rejection, as it might apply to amended Claims 7-9 and 45-48, is traversed for the following reasons. Claim 10 has been canceled.

Claims 7-9 and 45-48 require less than 3.0 milliequivalents of metathesis catalyst poison(s) per kilogram of fatty acid or fatty acid ester composition. The Office Action points to disclosure of sealing the fatty acid/ester under an inert atmosphere, such as nitrogen or argon, thereby concluding that the fatty acid/ester contains no poisons. The conclusion is flawed for the following reasons:

(1) The cited art discloses the fatty acid/ester composition in the context of a metathesis processes using metathesis catalysts, which the skilled person knows are sensitive to atmospheric moisture and oxygen. The cited art use of inert atmospheres protects the metathesis catalyst from inactivation by these atmospheric elements. The use of an inert atmosphere, however, during metathesis discloses nothing about metathesis catalyst poisons, such as organic hydroperoxides or water, which were already present in the fatty acid/ester feedstock fed to the metathesis process.

(b) The cited art does not illuminate the exposure of the unsaturated fatty acid/ester feedstock to metathesis catalyst poisons as may have been acquired from its commercial source or during handling and transit. Sealing the composition under an inert atmosphere does not purify an already contaminated feedstock. On the contrary, a decrease in catalyst poisons is effected by pre-treating the unsaturated fatty acid/ester feedstock over

Appl. No. 10/508,805  
Response B dated August 29, 2007  
Reply to Office Action of June 7, 2007

an adsorbent. Since the art says nothing also about pre-treatment of the feedstock over an adsorbent, no purification of the composition is implicitly disclosed. (WO'289 mentions purification of the metathesis product at page 15; but this is irrelevant. The invention is drawn to a composition intended as a feedstock to a metathesis process, not a product thereof.)

The Office Action alleges that the comparative data presented by Applicants are unconvincing and no unexpected or unobvious properties can be attributed to the claimed composition. Applicants respectfully request reconsideration.

In particular, the Office Action questions the data, because the "[t]he large amounts of catalyst referred by the Applicants fall within the ranges taught in the instant invention." This misses the point of the comparison presented. Applicants are not claiming the quantity of catalyst used or claiming the metathesis catalyst composition. Rather, the present invention concerns the effect of unsaturated fatty acid/ester feed purity on metathesis catalyst activity and lifetime, as measured by catalyst turnover number (CTN). This correlation is not contemplated in the cited references. A comparison has been made from data taken in the specification and the references.

As supported in Applicants' Examples E-1, E-2, and CE-1, lower concentrations of metathesis catalyst poisons in the fatty acid/ester fed to the metathesis process correlate with higher CTN. Moreover, Applicants' catalyst achieves about 10 times greater CTN as compared with the prior art catalysts. Thus, if the Examiner would review again the Table at page 16 in Applicants' Response A, filed September 22, 2006, it will be seen that Applicants' examples employ a lower concentration of catalyst (MO feed/Cat = 4500/1) while achieving a higher CTN (2,160-2,565), as compared with the cited references (MO feed/Cat = 200-600/1 for CTN 162-464).

The lower CTN of the prior art indicates that their catalysts are less active (partially deactivated) resulting from an un-named deactivating source, which consequentially leads the prior art to use a higher catalyst concentration to compensate for the deactivation. In contrast, Applicants' catalyst, with its higher activity and longer lifetime, e.g., less deactivation, can be used in lower concentrations, which is economically more advantageous when the catalyst uses expensive metals. The point of Applicants' discovery is this: the claimed feed purity directly correlates with higher catalyst activity and longer lifetime. Thus, the claimed fatty acid/ester compositions having less than 3.0 meq

Appl. No. 10/508,805  
Response B dated August 29, 2007  
Reply to Office Action of June 7, 2007

poison(s) per kg do possess unexpected properties in that they are beneficially more benign and less destructive towards metathesis catalysts.

In view of the above, the references do not support a disclosure, suggestion, or hint of the purity of the fatty acid/ester composition fed to the metathesis process, let alone the claimed purity of less than 3.0 meq metathesis catalyst poisons per kg composition. The references are not inherently anticipatory; they are unambiguously silent and do not address the issue at all. Accordingly, it is submitted that Claims 7-9 and 45-48 are novel; and it is respectfully requested that the rejection under 35 U.S.C. 102(b) be withdrawn.

**D. Regarding the Rejection of Claims 11-42, 49-51 under 35 USC 103(a)**

Claims 11-42 and 49-51 stand rejected under 35 USC 103(a) as being unpatentable over WO 96/04289. The Office Action asserts that it would have been obvious to use "fatty acids/esters derived from a seed oil" inasmuch as single fatty acid/esters, such as oleic acid and methyl oleate, are disclosed in an analogous process in WO'289; therefore, use of mixtures of fatty acid/esters derived from seed oils would be an obvious variation. This rejection, as it might apply to amended Claims 11-42 and 49-51, is traversed for the following reasons.

The Office Action misconstrues the invention; clarification is needed. Seed oils can be processed to provide for a single unsaturated fatty acid/ester, as in oleic acid or methyl oleate, or a mixture of unsaturated fatty acids or esters, as in vegetable oil blends typically of C<sub>14-22</sub> unsaturated fatty acids or esters. The invention is drawn towards using a single unsaturated fatty acid/ester derived from a seed oil or a mixture thereof derived from a seed oil, as found in the claim wording, "*a feedstock composition derived from a seed oil comprising one or more unsaturated fatty acids or unsaturated fatty acid esters.*" The number of fatty acid/esters in the feedstock is not the salient point.

The invention pertains to the purity of the unsaturated fatty acid/ester feedstock fed to the metathesis process and the effect of feed purity on metathesis catalyst activity and lifetime. As amended, Claims 11, 24, 33, and 39 require the fatty acid/ester feedstock to contain less than 25 meq metathesis catalyst poison(s) per kg composition so as to enable the metathesis catalyst preferably to achieve a catalyst turnover number (CTN) greater than 1,500 with use of a preferred C<sub>2-3</sub> olefin. WO'289 discloses nothing concerning how selected olefins and purity level of the fatty acid/ester feedstock correlates with CTN, let alone the specific limitations claimed.

Appln. No. 10/508,805  
Response B dated August 29, 2007  
Reply to Office Action of June 7, 2007

Aside from the aforementioned unobviousness, Claims 24 to 28 further relate to a process of preparing a reduced chain polyester polyepoxide that requires (a) metathesis, (b) (trans)esterification, and (c) epoxidation. Claim 29-30 and 31-32 are drawn to reduced-chain polyester polyolefin and polyester polyepoxide compositions, respectively, having chain lengths ranging from 6 to 10 carbon atoms ("x" in the formulas ranges from 3 to 7). Applicants can find no discourse or suggestion anywhere in WO'289 referring to transesterification of a reduced-chain metathesis product to yield a polyester polyolefin and epoxidation thereof to form a polyester polyepoxide. The claimed compositions are not envisioned at all. Thus, the skilled person can find no information, not even a thread, to arrive at the claims starting from WO'289; and thus, Claims 24-32 are clearly unobvious.

More to the point, Claims 33 to 36 relate to a process of preparing an  $\alpha,\omega$ -hydroxy acid,  $\alpha,\omega$ -hydroxy ester, or  $\alpha,\omega$ -diol comprising (a) metathesis and (b) hydroformylation with reduction. In Claim 36, the reduced chain  $\alpha,\omega$ -hydroxy acid or hydroxy ester is (c) (trans)esterified to yield an  $\alpha,\omega$ -polyester polyol. Claims 37-38 relate to a reduced chain  $\alpha,\omega$ -polyester polyol resulting in chain lengths of 7 to 11 carbon atoms (x in the formula ranges from 3 to 7). Applicants find no discourse or suggestion in WO'289 pertaining to (b) hydroformylation with reduction or (c) trans(esterification). Moreover, the claimed composition is nowhere envisioned. Accordingly, the skilled person cannot arrive at the claims starting from WO'289; Claims 33 to 36 are clearly unobvious.

Likewise, Claims 39 to 42 further relate to a process of preparing an  $\alpha,\omega$ -amino acid,  $\alpha,\omega$ -amino ester, or  $\alpha,\omega$ -diamine comprising (a) metathesis and (b) hydroformylation with reductive amination. In Claim 42, the  $\alpha,\omega$ -amino acid or amino ester is (c) (trans)esterified to yield an  $\alpha,\omega$ -polyester polyamine. No discourse or suggestion can be found in WO'289 for (b) hydroformylation of a metathesis product with reductive amination and (c) trans(esterification) to yield an  $\alpha,\omega$ -polyester polyamine; therefore, no basis exists for the skilled person to arrive at the claims starting from WO'289. Claims 39-42 are clearly unobvious.

Finally, we mention that present day commercial processes for preparing industrially useful chemicals, e.g., polyolefins, polyepoxides, polyesters, polyols, and polyamines, employ petroleum as a starting feedstock. Petroleum resources are dwindling in face of increased global demand. Unsaturated fatty acids/esters derived from seed oils could provide renewable, alternative feedstocks that could, at least partially, replace petroleum; however, the molecular weights of unsaturated fatty acids/esters (typically  $C_{14-30}$ ) make them

Appl. No. 10/508,805  
Response B dated August 29, 2007  
Reply to Office Action of June 7, 2007

inconvenient to handle. Applicants have discovered a new route to industrially useful reduced chain (about  $C_{7-11}$ ) polyester polyolefins, polyester polyepoxides, polyester polyols, and polyester polyamines of lower molecular weight for easier handling, via (a) metathesis of fatty acid/esters derived from seed oils and downstream functionalization and (trans)esterification of the reduced chain metathesis products. More importantly, Applicants have discovered that the seed oil feedstock to the metathesis process must possess the claimed purity, if the metathesis catalyst is to have a lifetime acceptable for commercial implementation, roughly at least 1,500 CTN.

In view of the above, it is submitted that all of Claims 11-42 and 49-51 meet the requirements for non-obviousness. It is respectfully requested that the rejection be withdrawn.

**E. Consideration of Correction of Inventorship**

In view of the cancellation of Claims 1-6 and 10 in this application, the undersigned has reviewed the inventorship of the pending claims and has determined that no correction of inventorship is required.

**F. Concerning the RCE and Supplementary Information Disclosure Statement**

On February 8, 2007, Applicants filed a Response to Final Rejection following receipt of a Final Rejection dated December, 14, 2006, in this application. Prior to a statutory deadline of June 14, 2007, Applicants filed on June 1, 2007, a Request for Continued Examination (RCE) with Supplementary Information Disclosure Statement. On June 7, 2007, the Patent Office responded to the submission of February 8, 2007, withdrawing the Final Rejection and issuing a new Non-Final Office Action to which Applicants have responded herein.

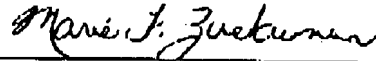
Applicants herein respectfully request the Examiner to consider the Supplementary Information Disclosure Statement (SIDS) submitted with the RCE. To that effect, Applicants provide herewith English-translations of the following documents cited in the SIDS: JP 62-83036 and RU 291908.

Appln. No. 10/508,805  
Response to dated August 29, 2007  
Reply to Office Action of June 7, 2007

**G. Conclusions**

Applicants gratefully acknowledge the allowability of Claims 43 and 44. In view of the amendments and arguments filed herein, Applicants believe that amended Claims 7-9 and 11-42 and 45-51 also meet all of the statutory requirements for patentability. A Notice of Allowance is respectfully solicited at the Examiner's earliest convenience.

Respectfully submitted,



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Enc. (2 translations)

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## TRANSLATION

(19) Japan Patent Office (JP)

(11) Patent Application Kokai

No. S62-83036

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4/16/1987

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	13/00	7215 - 4H

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Examination Request: Not requested Number of claims: 1 (3 pages)

(54) Title of the Invention : Adsorption Assist Agent and the Adsorption Agent That Contains Said Adsorption Assist Agent

(21) Application No. : S60-224103

(22) Application Date : Oct. 8, 1985

(72) Inventor : Morio Ueno? (*Spelling uncertain, Translator*)  
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Description

## 1. Title of the Invention :

Adsorption Assist Agent and the Adsorption Agent That Contains Said  
Adsorption Assist Agent

## 2. Scope of the Patent Application

## Claim 1

Adsorption assist agent made with organic acid salt compounds, etc., which is comprised of bonding of one or two or more organic acids such as a carboxylic acid, ascorbic acid, etc., and one or two of calcium and magnesium ions.



No. S62-83036

**Claim 2**

Adsorption agent made with organic acid salt compounds, etc., which is comprised of bonding of one or two or more organic acids such as a carboxylic acid, ascorbic acid, etc., and one or two of calcium and magnesium ions, and one or two of sodium and potassium ions.

**Claim 3**

Adsorption assist agent which contains the adsorption assist agent made with the organic acid salt compounds of one or both of the above mentioned Claim 1 and Claim 2, and one or two of silicate and aluminum salt compounds.

**Claim 4**

Adsorption assist agent which contains one or two of the adsorption assist agents described in the above mentioned Claims 1, 2 and 3, and one or two phosphate compounds.

**Claim 5**

Adsorption agent which contains one or two or more of the adsorption assist agents described in the above mentioned Claims 1, 2, 3 and 4, and one or two or more adsorption agents such as activated charcoal, bone char, activated white clay, acidic white clay, pearlite, zeolites and the composition that has an adsorbing function.

**3. Detailed Explanation of the Invention****[Field of Utilization in Industry]**

This invention relates to the adsorption assist agent and the adsorption agent that contains said adsorption assist agent which can exhibit an adsorbing function, even in a high temperature zone, without decreasing the adsorbing functions such as de-coloring, de-oxygenation, peroxide removal, odor removal, etc., for oils by co-using the adsorption agents and adsorption assist agents that have been used until now.

No. S62-83036

**[Existing Technology]**

Concerning the regeneration of the waste oil from the oils and lipids, the method in which the waste oil is mixed with an organic solvent and it is cooled to -30 ~ -40 °C, and the un-polymerized part and the polymerized part are separated (Patent Kokoku No. S58-25400); the method in which the oxidation rate of the oil is slowed down by submerging (wheat rice rock *(this whole text is hand written, and it looks like Awheat rice®, Translator)*) into the oil (Patent Kokoku No. S57-26100); the method in which the oxidation rate of the oil is slowed down by covering the metal surface of the heating container and by submerging the wheat rice rock (patent Kokai No. S58-162248); the method of using a precise filtering machine as the de-coloring method; the method in which the deterioration of the oil is slowed down by co-using oil and water in the fryers; the method in which the oxidation rate is slowed down by utilizing electricity to charge a superposition wave; the method which uses the oxides, hydroxides and carbonates of calcium and magnesium; calcium silicate; magnesium silicate; aluminum hydroxide gel, etc., as the de-oxidation agents, have been reported, however, this de-oxidation temperature zone is low, around 80 ° C, and in high temperature zones, the color increases and also the de-oxygenation ability decreases. In addition, there is the oil purification method which has been used until now as the normal method, but all of these are low temperature treatments.

**[Problem That this Invention Intends to Solve]**

In the above mentioned purification methods or the deterioration prevention methods or the regeneration methods, the treatment processes for de-coloring, de-oxygenation, peroxide removal, odor removal, are all done individually, and this is a nuisance from a process standpoint. Also, some adsorption agents for de-coloring, can increase the deterioration of oils and decrease the de-coloring ability at high temperatures, therefore, if the de-coloring, de-oxygenation, peroxide removal and odor removal are performed by utilizing the existing methods, the deterioration of the oil might occur too and this is a problem.

No. S62-83036

**[Method to Solve the Problem]**

The above mentioned existing problems were solved by the application No. 60-200285 : AComposition that has an adsorbing function and the Patent Application on Sept. 30, 1985, AAdsorption assist agent and adsorption agent that contains said adsorption assist agent. Thereafter, into the adsorption agents that have been used until now, when the adsorption assist agent of an organic acid salt compound, etc., of this invention, or the adsorption assist agent that contains an adsorption assist agent of an organic acid salt compound and one or two or more of silicates and aluminum salts, etc., or the adsorption assist agent which contains one or two or more of the above mentioned adsorption assist agents, and one or two or more phosphate compounds, or the adsorption agent which contains one or two or more of the above mentioned adsorption assist agents, and one or two or more adsorption agents such as activated charcoal, bone char, activated white clay, acidic white clay, pearlite, zeolite and the composition that has an adsorbing function, is used, the treatment of oils such as de-coloring, de-oxygenation, peroxide removal, odor removal, etc., can be done in the high temperature zone, and thus the problems are solved.

**[Action]**

When the above mentioned adsorption assist agents are co-used with the adsorption agents that have been used until now, not only can the de-coloring, de-oxygenation, peroxide removal; odor removal, etc., be done in the low temperature zone and the high temperature zone (50 ~ 200 °C), but also this nature is utilized, and when the oil that is being used at high temperature for cooking is circulated, while being contacted with the adsorption agent that contains an adsorption assist agent, de-coloring, de-oxygenation, peroxide removal, odor removal, etc., is performed, so that the deteriorated material in the oil is less, and it maintains this state, this prevents the progress of deterioration which progresses by an auto catalytic reactive mechanism. *(the meaning of this part is unclear, is the deterioration an auto catalytic reaction? or is prevention an auto catalytic reaction?, Translator)* Also, in the regeneration of oils, they can be regenerated to near the state of new oils. Also, it can be co-used with one of the de-oxygenation agents, etc., which have been reported until now too.

No. S62-83036

**[Actual Examples]**

Next, the compounds that are used in this invention as the organic acids such as carboxylic acids; calcium and magnesium ions; sodium and potassium ions; silicate; aluminum salt compound, will be explained.

**1. Carboxylic acids**

These are organic compounds that have a carboxylic group -COOH, and there are mono carboxylic acids, di-carboxylic acids and tri carboxylic acids. Depending on the hydrocarbon group, there are chain style carboxylic acids, aromatic carboxylic acids, saturated carboxylic acids, unsaturated carboxylic acids. For example, there is citric acid; gluconic acid; acetic acid; oxalic acid; tartaric acid; lactic acid; malic acid; fumaric acid; alginic acid; propionic acid; pantothenic acid; etc.

**2. Calcium and magnesium ion compounds****< Calcium compounds >**

Calcium carbonate; calcium hydroxide; calcium oxide; calcium chloride;

**< Magnesium compounds >**

Magnesium carbonate; magnesium hydroxide; magnesium oxide; magnesium chloride; magnesium sulfate

**3. Sodium and potassium ion compounds****< Sodium compounds >**

Sodium carbonate; sodium bicarbonate; sodium peroxide; sodium phosphate; sodium silicate; sodium hydroxide;

**< Potassium compounds >**

Potassium carbonate; potassium bicarbonate; potassium hydroxide; potassium phosphate; potassium oxide; potassium silicate; potassium phosphate

No. S62-83036

**4. Aluminum salt compounds**

Aluminum sulfate; alum powder; aluminum hydroxide; aluminum gel;

**5. Silicates**

Compounds that can be indicated by  $xM^{1}_{2}O$   $YxSiO_{2}$ , water containing salts, composite salts, also the ones of which form contains a negative atomic group such as an acid group, etc., for instance, almino silicates, etc., that contains  $Al_{2}O_{3}$ .

M = Al, Fe, Ca, Mg, Na, K, etc.

Next, the adsorption assist agent made with an organic acid salt compound, and a phosphate compound adsorption assist agent will be explained.

**< Organic acid salt compounds >**

Calcium oxalate; magnesium oxalate;

Calcium tartrate, magnesium tartrate;

Calcium acetate, magnesium acetate;

Calcium gluconate, magnesium gluconate,

Calcium lactate; magnesium lactate;

Calcium citrate; magnesium citrate;

Calcium malate; magnesium malate;

Calcium ascorbate; magnesium ascorbate

Calcium propionate; magnesium propionate; calcium pantothenate

**< Phosphate compounds >**

Ca  $(Na_{2}PO_{4})_{2}$   $CaNaPO_{4}$ .

$Ca_{3}(PO_{4})_{2}$ ;  $Ca(H_{2}PO_{4})_{2}$ ;

$CaHPO_{4}$ ;  $Ca(MgPO_{4})_{2}$

Mg  $(Na_{2}PO_{4})_{2}$ ;  $MgNaPO_{4}$ ;

$Mg_{3}(PO_{4})_{2}$ ;  $Mg(CaPO_{4})_{2}$

$Ca(K_{2}PO_{4})_{2}$ ;  $Mg(K_{2}PO_{4})_{2}$

Based on the above mentioned representative examples, the actual example of this

No. S62-83036

invention will be explained.

#### <Actual Example >

An adsorption assist agent which was comprised of 10 parts of calcium citrate, 10 parts of aluminum silicate and 10 part of aluminum sulfate; and an adsorption agent that was comprised of 50 parts of activated white clay and 20 parts of activated charcoal, were added at 5 % into the tempura cooking oil at 180 °C, and after it was mixed, it was filtered, and the results for the de-coloring, de-oxygenation, peroxide removal, odor removal, etc., of this tempura oil are shown in Table 1.

Table 1.

	Nature of the purified oil				
		Color level Y / R	Acid value	Peroxide value	Odor
number of purifications performed	0	51 / 4.1	2.8	12.1	++
	1	15 / 1.3	1.2	6.8	-
	2	4 / 0.6	0.03	3.7	-

Color level : Lovibond color level 24.5 mm cell was used.

Odor :

++ smells strongly.

+ smells a little

- does not smell.

#### < Effect of the Invention >

As was mentioned above, this invention is the adsorption assist agent and the adsorption agent that contains said adsorption assist agent which can perform de-coloring, de-oxygenation, peroxide removal, odor removal, etc., at the same time in the low temperature zone and even in the high temperature zone, in the purification process, the regeneration process and the deterioration prevention processes for oils, and it is economically advantageous by conserving the energy in the purification process of oils. Also, the adsorption assist agent and the adsorption agent that contains the adsorption assist agent, can be used even in the high temperature zone, so that deterioration of oil during cooking can be prevented, and the product quality can

No. S62-83036

be stabilized, and this is an advantage. Also, even the oil that became waste oil can be regenerated via regeneration treatment until the nearly new oil state and it can be re-used, and that is an advantage too.

Patent Applicant : Morio Ueno?

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The Union of the Soviet Socialist Republics  [USSR State Emblem]  The State Committee of USSR Ministers' Council on Inventions and Discoveries	<b>INVENTION DESCRIPTION</b> <b>TO AUTHOR CERTIFICATE</b> Derivative from the Author Certificate # _____ Claim on 08/15/1969 (#1360503/23-4) With addition of claim # _____ Priority _____ Published on 01/06/1971, Bulletin # 4 Description publication date 03/02/1971	<b>291908</b>  IPC C 07 c 51/00 C 07 c 57/12  UDC 547.392.4 (088.8)
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Invention Authors

M.S. Levit, Z.P. Kazachkova, N.D. Klochko, S.A. Vetkazova  
and D.M. Makarin

Claimant

All-Union Research and Development Institute of Fats

**TECHNICAL OLEIC ACID PRODUCTION METHOD**

The proposed product can be used for production of synthetic fiber (lavan).

The method of technical oleic acid production from vegetable oils is known using their primary purification with table salt solution at the temperature of 60-80°C, neutralization by caustic soda, and disintegration at the temperature of 220-250°C and under pressure 24-25 at with subsequent extraction of fatty acids and their mixing.

The obtained technical oleic acid comprises of no more than 75% of pure oleic acid and up to 17% of unsaturated fatty acids C15 similar to linoleic and linolenic acids with two and three double bonds producing a negative impact on fiber treatment.

In addition to it, this technical oleic acid is susceptible to auto-oxidation due to high content of linoleic and linolenic acids, so it needs using inhibitors for stabilization at storing.

The objective of the described invention is enlarging of raw material source and improvement of a target product quality.

This objective is achieved by using a mixture of mono-ene and di-ene acids produced by fractionating of sperm whale body fat acids (after extracting alcohols from them) with vacuum distillation units at the temperature of 210°C and residual pressure 10-15 mm of a mercury column. After distillation of an upper fraction (its amount is 48-54% of the initial raw material), a bottom fraction was treated with additional distillation (in order to produce a light-colored product) at the temperature of 225°C and residual pressure 10-15 mm of a mercury column.

The obtained second fraction represents a target product. Its yield is 40-42% of the initial raw material.

The proposed technical oleic acid contains:

- a) 70-75% unsaturated mono-ene fatty acids C14, C16, C18, C20, C22 (including 37% of oleic acid, 10% of palmitoleic acid C16, 1-2% of myristoleic acid, 28% of C20, and 2-3% of C22);



- b) 2-11% of di-ene fatty acids (including 2% of linoleic, 90% of C20, C22);  
 c) the rest, up to 100%, are saturated fatty acids (including stearic, palmitoleic, myristoleic, and C20 acids).

This composition provides better characteristics of stability and color due to absence in the product poly unsaturated fatty acids.

Comparative physical and chemical characteristics of the oleic acid produced in the Soviet Union, foreign sample, and the proposed product are shown in a table.

Table

Items	Characteristics and norms		
	Grade "B" olein, GOST 7580-50	Foreign sample	Oleic acid analog (proposed)
Appearance	Liquid	Liquid	Liquid
Color	Yellow	Light yellow	Light yellow
Acid number	185 - 200	200 - 206	170 - 200
Saponification number	185 - 200	200 - 206	173 - 203
Iodine number, in g of iodine per 100 g	85 - 105	88 - 92	80 - 90
Unsaponifiable matter content, %	2.5	2.5	1.5
Mono-ene unsaturated fatty acids content, %	63 - 74	73 - 75	70 - 75
Di-ene and tri-ene acids in type similar to linoleic and linolenic acids content, %	17	8.5 - 11.5	2 - 11
Saturated fatty acids content, %	8 - 10	12 - 14	12 - 15

Example. 10 kg of sperm whale body fat acids (after extraction of alcohols) were undergone fractionating on a laboratory distillation unit. The upper volatile fraction containing saturated and unsaturated acids C14 and C16 is used in soap production, and a bottom residue is treated with additional distillation in order to remove coloring, oxi-fatty and polymerized acids. There were obtained 4.1 kg of distilled fatty acids of light yellow color representing an analog of oleic acid.

#### Invention Formula

The method of technical oleic acid production distinctive in that, with an objective of enlarging of raw material source and improvement of a target product quality, sperm whale body fat acids are treated with subsequent distillation at the temperature up to 210°C and residual pressure 10-15 mm of a mercury column, and the remaining after that fraction is treated with distillation at 225°C and the same pressure.